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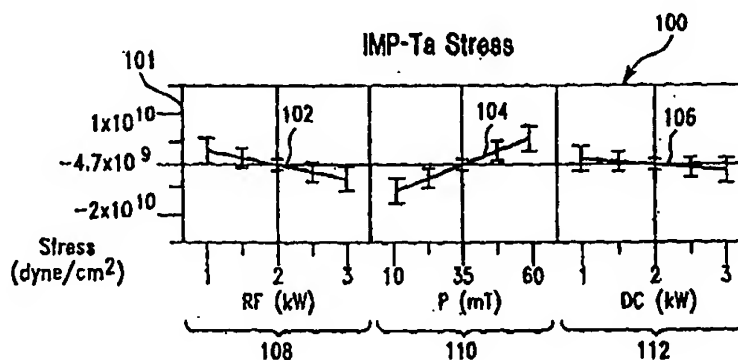
(57) Abstract

We have discovered that the residual stress residing in a tantalum (Ta) film or a tantalum nitride (TaN$_x$, where $0 < x \leq 1.5$) film can be controlled (tuned) by controlling particular process variables during deposition of the film. Process variables of particular interest during film deposition, for sputter–applied Ta and TaN$_x$ films, include the following: the power to the sputtering target; the process chamber pressure (*i.e.*, the concentration of various gases and ions present in the chamber); the substrate DC offset bias voltage (typically an increase in the AC applied substrate bias power); and the temperature of the substrate upon which the film is being deposited. When the Ta or TaN$_x$ film is deposited using IMP sputtering, the power to the ionization coil can be used for stress tuning of the film. This use of IMP as the sputtering technique provides particular control over the ion bombardment of the depositing film surface. Tantalum (Ta) films deposited using the IMP method typically exhibit a residual stress ranging from about $+1 \times 10^{+10}$ dynes/cm^2 (tensile stress) to about $-2 \times 10^{+10}$ dynes/cm^2 (compressive stress), depending on the process variables described above. Tantalum nitride (TaN$_x$) films deposited using the IMP method typically can be tuned to exhibit a residual stress within the same range as that specified above with reference to Ta films. We have been able to reduce the residual stress in either the Ta or TaN$_x$ films to range between about $6 \times 10^{+9}$ dynes/cm^2 and about $-6 \times 10^{+9}$ dynes/cm^2 using tuning techniques described herein. The Ta and TaN$_x$ films can also be tuned subsequent to deposition using ion bombardment of the film surface and annealing of the deposited film. Barrier performance of the films can be improved by depositing the films at a substrate temperature of at least 300 °C.

1   **STRESS TUNABLE TANTALUM AND TANTALUM NITRIDE FILMS**

2   **BACKGROUND OF THE INVENTION**

3   1.   Field of the Invention

4   The present invention pertains to tantalum and tantalum nitride films which can

5   be stress-tuned to be in tension or in compression or to have a particularly low stress, and

6   to a method of producing such films. These stress-tuned films are particularly useful in

7   semiconductor interconnect structures, where they can be used to balance the stress

8   within a stack of layers, which includes a combination of barrier layers, wetting layers,

9   and conductive layers, for example. The low stress tantalum and tantalum nitride films

10   are particularly suited for the lining of vias and trenches having high aspect ratios.

11   2.   Brief Description of the Background Art

12   A typical process for producing a multilevel structure having feature sizes in the

13   range of 0.5 micron (μm) or less would include: blanket deposition of a dielectric

14   material; patterning of the dielectric material to form openings; deposition of a diffusion

15   barrier layer and, optionally, a wetting layer to line the openings; deposition of a

16   conductive material onto the substrate in sufficient thickness to fill the openings; and

17   removal of excessive conductive material from the substrate surface using chemical,

18   mechanical, or combined chemical-mechanical polishing techniques. Future

19   technological requirements have placed a focus on the replacement of aluminum (and

20   aluminum alloys) by copper as the conductive material. As a result, there is an increased

21   interest in tantalum nitride barrier layers and in tantalum barrier/wetting layers, which

22   are preferred for use in combination with copper.

1   Tantalum nitride barrier films, Ta_2N and TaN, have been shown to function up to

2   700°C and 750°C, respectively, without the diffusion of copper into an underlying

3   silicon (Si) substrate. Tantalum barrier/wetting films have been shown to function at

4   temperatures of approximately 500°C. It is advantageous in terms of processing

5   simplicity to sputter the barrier and/or wetting layers underlying the copper. Tantalum

6   nitride barrier layers are most commonly prepared using reactive physical sputtering,

7   typically with magnetron cathodes, where the sputtering target is tantalum and nitrogen

8   is introduced into the reaction chamber.

9   S. M. Rossnagel et al. describe a technique which enables control of the degree of

10   directionality in the deposition of diffusion barriers in their paper titled "Thin, high

11   atomic weight refractory film deposition for diffusion barrier, adhesion layer, and seed

12   layer applications", *J. Vac. Sci. Technol.* B 14(3) (May/Jun 1996). In particular, the

13   paper describes a method of depositing tantalum (Ta) which permits the deposition of the

14   tantalum atoms on steep sidewalls of interconnect vias and trenches. The method uses

15   conventional, non-collimated magnetron sputtering at low pressures, with improved

16   directionality of the depositing atoms. The improved directionality is achieved by

17   increasing the distance between the cathode and the workpiece surface (the throw) and

18   by reducing the argon pressure during sputtering. For a film deposited with commercial

19   cathodes (Applied Materials Endura® class; circular planar cathode with a diameter of

20   30 cm) and rotating magnet defined erosion paths, a throw distance of 25 cm is said to be

21   approximately equal to an interposed collimator of aspect ratio near 1.0. In the present

22   disclosure, use of this "long throw" technique with traditional, non-collimated magnetron

23   sputtering at low pressures is referred to as "gamma sputtering".

24   Gamma sputtering enables the deposition of thin, conformal coatings on

25   sidewalls of a trench having an aspect ratio of 2.8 : 1 for 0.5 μm wide trench features.

26   However, we have determined that gamma-sputtered TaN films exhibit a relatively high

1   film residual compressive stress, in the range of about -1.0 x 10^{+10} to about

2   -5.0 x 10^{+10} dynes/cm^2. High film residual compressive stress, in the range described

3   above, can cause a Ta film or a tantalum nitride (*e.g.*, Ta_2N or TaN) film to peel off from

4   the underlying substrate (typically silicon oxide dielectric). In the alternative, the film

5   stress can cause feature distortion on the substrate (typically a silicon wafer) surface or

6   even deformation of a thin wafer.

7   A method of reducing the residual stress in a Ta barrier/wetting film or a Ta_2N or

8   TaN barrier film would be beneficial in enabling the execution of subsequent process

9   steps without delamination of such films from trench and via sidewalls or other

10   interconnect features. This reduces the number of particles generated, increasing device

11   yield during production. In addition, a film having a near zero stress condition improves

12   the reliability of the device itself.

13   **SUMMARY OF THE INVENTION**

14   We have discovered that the residual stress residing in a tantalum (Ta) film or a

15   tantalum nitride (TaN_x, where $0 < x \leq 1.5$) film can be controlled (tuned) by controlling

16   particular process variables during deposition of the film. Process variables of particular

17   interest for sputter-applied Ta and TaN_x films include the following: An increase in the

18   power to the sputtering target (typically DC) increases the compressive stress component

19   in the film. An increase in the process chamber pressure (*i.e.*, the concentration of

20   various gases and ions present in the chamber) increases the tensile stress component in

21   the film. An increase in the substrate DC offset bias voltage (typically an increase in the

22   applied AC as substrate bias power) increases the compressive stress component in the

23   film. When the sputtering is IMP sputtering, an increase in the power to the ionization

24   coil increases the compressive stress component in the film. The substrate temperature

25   during deposition of the film also affects the film residual stress. Of these variables, an

1   increase in the process chamber pressure and an increase in the substrate offset bias most

2   significantly affect the tensile and compressive stress components, respectively.

3   The most advantageous tuning of a sputtered film is achieved using Ion Metal

4   Plasma (IMP) sputter deposition as the film deposition method. This sputtering method

5   provides for particular control over the ion bombardment of the depositing film surface.

6   When it is desired to produce a film having minimal residual stress, particular care must

7   be taken to control the amount of ion bombardment of the depositing film surface, as an

8   excess of such ion bombardment can result in an increase in the residual compressive

9   stress component in the deposited film.

10   Tantalum (Ta) films deposited using the IMP sputter deposition method typically

11   exhibit a residual stress ranging from about $+1 \times 10^{+10}$ dynes/cm^2 (tensile stress) to about

12   $-2 \times 10^{+10}$ dynes/cm^2 (compressive stress), depending on the process variables described

13   above. Tantalum nitride (TaN$_x$) films deposited using the IMP method typically can be

14   tuned to exhibit a residual stress within the same range as that specified above with

15   respect to Ta films. We have been able to reduce the residual stress in either the Ta or

16   TaN$_x$ films to low values ranging from about $+1 \times 10^{+9}$ to about $-2 \times 10^{+9}$ dynes/cm^2

17   using tuning techniques described herein. These film residual stress values are

18   significantly less than those observed for traditionally sputtered films and for gamma-

19   sputtered films. This reduction in film residual compressive stress is particularly

20   attributed to bombardment of the film surface by IMP-generated ions during the film

21   deposition process. Heavy bombardment of the film surface by IMP-generated ions can

22   increase the film residual compressive stress, so when it is desired to minimize the film

23   compressive stress, the ion bombardment should be optimized for this purpose.

24   Other process variables which may be used in tuning the film stress include the

25   spacing between the sputter target and the substrate surface to be sputter deposited; ion

26   bombardment subsequent to film deposition; and annealing of the film during or after

4

1   deposition.

2   We have also discovered that Ta and TaN$_x$ films deposited using physical vapor

3   deposition techniques over a particular temperature range have improved diffusion

4   barrier properties. The lower end of the temperature range is approximately 300°C.

5   Using a deposition temperature of 300°C or higher, the barrier layer can be thinner,

6   leading to better manufacturing through-put. Further, a thinner barrier layer requires less

7   chemical-mechanical polishing to remove residual barrier layer from a substrate surface

8   surrounding a copper interconnect, and this reduces the amount of copper which is

9   removed from the interconnect due to the polishing action (copper "dishing").

10   Accordingly, disclosed herein is an improved method of depositing a Ta or TaN$_x$ barrier

11   layer for use in combination with copper in a semiconductor interconnect structure. The

12   improvement comprises depositing a Ta or TaN$_x$ (where $0 < x \leq 1.5$) barrier layer at a

13   substrate temperature of at least 300°C, preferably, within a temperature range of about

14   300°C to about 600°C, most preferably, within the range of about 300°C to about

15   500°C, prior to deposition of copper on the substrate.

16   **BRIEF DESCRIPTION OF THE DRAWINGS**

17   Figure 1 is a graph showing the residual stress in an IMP-deposited Ta film, as a

18   function of DC power to the Ta target, RF power to the IMP ionization coil, and the

19   pressure in the process chamber.

20   Figure 2A is a contour plot showing the IMP-deposited Ta film residual stress in

21   dynes/cm^2, as a function of the DC power to the Ta target and the process chamber

22   pressure, when the RF power to the ionization coil is 1 kW.

23   Figure 2B is a contour plot showing the residual stress in an IMP-deposited Ta

24   film, as a function of the same variables illustrated in Figure 2A, when the RF power to

25   the ionization coil is 3 kW.

1   Figure 3 is a graph showing the residual stress in an IMP deposited Ta film, as a

2   function of the substrate offset bias, and in particular as a function of the AC bias power

3   (typically the AC power is coupled to the substrate through the substrate heater which is

4   in electrical contact with the substrate).

5   Figure 4 is a graph showing the chemical composition of a gamma-sputtered

6   tantalum nitride film, as a function of the nitrogen gas flow rate to the sputtering process

7   chamber. In addition, Figure 4 shows the resistivity and the structure of the tantalum

8   nitride compound, which is in conformance with the nitrogen content of the compound.

9   Figure 5 is a graph showing the film composition of a reactive IMP-deposited

10   tantalum nitride film, as a function of the nitrogen gas flow rate to the process chamber.

11   Again, the resistivity of the film is indicative of the various film structures created as the

12   nitrogen content of the film is increased.

13   Figure 6 is a graph showing the residual film stress for gamma-sputtered tantalum

14   nitride film, as a function of the nitrogen gas flow rate to the sputtering process chamber,

15   and as a function of the temperature at which the film is deposited.

16   Figure 7 is a graph showing the residual film stress for reactive IMP sputtered

17   tantalum nitride film, as a function of the nitrogen gas flow rate to the sputtering process

18   chamber.

19   Figure 8 is a graph showing the residual film stress for IMP sputtered tantalum

20   film, as a function of the substrate temperature upon which the film is deposited.

1   **DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS**

2   The present invention pertains to stress tunable tantalum and tantalum nitride

3   films and to a method of producing such films. In particular, applicants have discovered

4   that residual film stress can be tuned by controlling particular process variables, such as

5   process chamber pressure, DC offset bias voltage, power to the sputtering target, and

6   substrate temperature during film deposition. When IMP sputtering is used, a variation

7   in the power to the ionization coil can be used for tuning. Ion bombardment of the

8   depositing film surface is particularly useful in controlling residual film stress.

9   I.   DEFINITIONS

10   As a preface to the detailed description, it should be noted that, as used in this

11   specification and the appended claims, the singular forms "a", "an", and "the" include

12   plural referents, unless the context clearly dictates otherwise. Thus, for example, the

13   term "a semiconductor" includes a variety of different materials which are known to have

14   the behavioral characteristics of a semiconductor, reference to a "plasma" includes a gas

15   or gas reactants activated by an RF glow discharge, and reference to "copper" includes

16   alloys thereof.

17   Film stress values were measured using a Tencor® Flexus FLX 3200 machine

18   available from Tencor Corporation, Mountain View, California.

19   Specific terminology of particular importance to the description of the present

20   invention is defined below.

21   The term "aspect ratio" refers to the ratio of the height dimension to the width

22   dimension of particular openings into which an electrical contact is to be placed. For

23   example, a via opening which typically extends in a tubular form through multiple layers

24   has a height and a diameter, and the aspect ratio would be the height of the tubular

25   divided by the diameter. The aspect ratio of a trench would be the height of the trench

1   divided by the minimal travel width of the trench at its base.

2   The term "completely filled" refers to the characteristic of a feature, such as a

3   trench or via, which is filled with a conductive material, wherein there is essentially no

4   void space present within the portion of the feature filled with conductive material.

5   The term "copper" refers to copper and alloys thereof, wherein the copper content

6   of the alloy is at least 80 atomic % copper.   The alloy may comprise more than two

7   elemental components.

8   The term "feature" refers to contacts, vias, trenches, and other structures which

9   make up the topography of the substrate surface.

10   The term "gamma or (γ) sputtered copper" refers to the "long throw" sputtering

11   technique described in the paper by S. M. Rossnagel et al., which was discussed

12   previously herein. Typically, the distance between the substrate and the target is about

13   the diameter of the substrate or greater; and, preferably, the process gas pressure is

14   sufficiently low that the mean free path for collision within the process gas is greater

15   than the distance between the target and the substrate.

16   The term "ion metal plasma" or "IMP" refer to sputter deposition, preferably

17   magnetron sputter deposition (where a magnet array is placed behind the target). A high

18   density, inductively coupled RF plasma is positioned between the sputtering cathode and

19   the substrate support electrode, whereby at least a portion of the sputtered emission is in

20   the form of ions at the time it reaches the substrate surface.

21   The term "IMP sputtered tantalum" refers to tantalum which was sputtered using

22   the IMP sputter deposition method.

23   The term "IMP sputtered tantalum nitride" refers to tantalum nitride which was

24   sputtered using the IMP sputter deposition method.

1   The term "reactive IMP sputtered tantalum nitride" refers to ion deposition

2   sputtering, wherein nitrogen gas is supplied during the sputtering of tantalum, to react

3   with the ionized tantalum, producing an ion deposition sputtered tantalum nitride-

4   comprising compound.

5   The term "stress-tuned" refers to a TaN_x or Ta film which has been treated during

6   processing to adjust the residual stress within the deposited film to fall within a particular

7   desired range. For example, at times it is desired to use the TaN_x or Ta film to balance

8   the overall stress within a stack of layers, so the film may be tuned to be in compression

9   or tension. At other times, it may be desired to reduce the stress in the film to be as near

10   to zero as possible.

11   The term "traditional sputtering" refers to a method of forming a film layer on a

12   substrate, wherein a target is sputtered and the material sputtered from the target passes

13   between the target and the substrate to form a film layer on the substrate, and no means is

14   provided to ionize a substantial portion of the target material sputtered from the target

15   before it reaches the substrate. One apparatus configured to provide traditional

16   sputtering is disclosed in U.S. Patent No. 5,320,728, the disclosure of which is

17   incorporated herein by reference. In such a traditional sputtering configuration, the

18   percentage of target material which is ionized is less than 10%, more typically, less than

19   1%, of that sputtered from the target.

20   II.   AN APPARATUS FOR PRACTICING THE INVENTION

21   A process system in which the method of the present invention may be carried

22   out is the Applied Materials, Inc. (Santa Clara, California) Endura® Integrated

23   Processing System. The system is shown and described in United States Patent No.

24   5,186,718, the disclosure of which is hereby incorporated by reference.

9

1   .   The traditional sputtering process is well-known in the art. The gamma

2   sputtering method is described in detail by S. M. Rossnagel et al. in their paper titled

3   "Thin, high atomic weight refractory film deposition for diffusion barrier, adhesion layer,

4   and seed layer applications", as referenced above. The IMP sputtering method is also

5   described by S. M. Rossnagel and J. Hopwood in their paper "Metal ion deposition from

6   ionized magnetron sputtering discharge, *J. Vac. Sci. Technol.* B, Vol. 12, No. 1 (Jan/Feb

7   1994).

8   III.   THE STRUCTURE OF THE TANTALUM AND TANTALUM NITRIDE
9   FILMS

10   We have been able to create a copper filled trench or via, which is completely

11   filled, at a feature size of about 0.4 μm and an aspect ratio of greater than 1 : 1 (up to

12   about 3 : 1 presently). To facilitate the use of a copper fill, the trench or via (constructed

13   in a silicon oxide surface layer) was lined with a reactive IMP-sputtered TaN$_x$ barrier

14   layer, followed by a Ta barrier/wetting layer, to create a bilayer over the oxide surface

15   layer. The copper fill layer was applied using a sputtering technique in the manner

16   described in Applicants' copending application, U.S. application Serial No. 08/855,059,

17   filed May 13, 1997, which is hereby incorporated by reference in its entirety.

18   In particular, the copper fill layer may be applied in a single step process or in a

19   two step process. In the single step process, for feature sizes of about 0.75 μm or less,

20   when the aspect ratio of the feature to be filled is less than approximately 3 : 1, the

21   temperature of the substrate to which the copper fill layer is applied should range from

22   about 200 °C to about 600 °C (preferably from about 200 °C to about 500 °C); when the

23   aspect ratio is about 3 : 1 or greater, the copper fill layer should be applied over a

24   temperature ranging from about 200 °C to about 600 °C (preferably from about 300 °C

25   to about 500 °C). The deposition can be initiated at the low end of the temperature

26   range, with the temperature being increased during deposition.

1   In the two step process, a thin, continuous wetting (bonding) layer of copper is

2   applied at a substrate surface temperature of about 20 °C to about 250 °C. The wetting

3   layer thickness (on the wall of the trench or via) should be a minimum of about 50 Å, and

4   typically may be about 100 Å to about 300 Å, depending on feature size and aspect ratio.

5   Subsequently, the temperature of the substrate is increased, with the application of fill

6   copper beginning at about 200 °C or higher and continuing as the temperature is

7   increased to that appropriate for the feature size. When both the copper wetting layer

8   and the copper fill layer are applied in a single process chamber, the deposition may be a

9   continuous deposition. In such case, process conditions are varied during the deposition,

10   with the copper fill layer being applied at a slower rate than the copper wetting layer, to

11   provide better deposition control.

12   When the copper wetting layer is applied in one process chamber and the copper

13   fill layer is applied in a second process chamber, typically the substrate with copper

14   wetting layer already applied is placed on a heated support platen in the second process

15   chamber. For a small feature size (0.5 μm or less) and an aspect ratio of 1 : 1 or greater,

16   it is better to wait until the substrate is heated to a temperature of at least 200 °C prior to

17   beginning application of the copper fill layer, or to begin the fill layer deposition at a

18   slower rate while the substrate is heating.

19   The copper sputtering technique used in the single step process is selected from

20   Gamma deposited copper, Coherent copper, IMP copper, and traditional standard sputter

21   deposition copper.

22   The copper deposition method used for application of the thin, continuous,

23   wetting layer of copper in the two step process may be one of the sputtered copper

24   techniques listed above or may be chemical vapor deposition (CVD) copper or

25   evaporation deposited copper, depending on the feature size of the trench or via to be

26   filled. The deposition method used for the copper fill layer is selected from the

1   sputtering techniques listed above, to provide a more contaminant-free and more rapid

2   filling of the trench or via.

3   To ensure the overall dimensional stability of the structure, we investigated

4   various factors which affect the residual film stress in a TaN_x barrier layer and in a Ta

5   layer (which can serve as a barrier layer, a wetting layer, or both, depending on the

6   application).

7   One skilled in the art can envision a combination of a number of different layers

8   underlying the copper fill material. Whatever the combination of layers, they provide a

9   stack of layers, and tuning the stress of individual layers within the stack can provide a

10   more stress-balanced and dimensionally stable stack. Although the preferred

11   embodiment described above is for the lining of trenches and vias, one skilled in the art

12   will appreciate that the stress-tuned TaN_x and Ta films described herein have general

13   application in semiconductor interconnect structures. The method of controlling and

14   reducing the residual film stress in tantalum nitride and tantalum films can be used to

15   advantage in any structure in which a layer of such a film is present. The concept of

16   tuning the residual stress in a sputter-deposited film comprising at least one metal

17   element has broad applicability.

18   IV.   THE METHOD OF TUNING RESIDUAL STRESS IN TANTALUM AND
19   TANTALUM NITRIDE FILMS

20   The preferred embodiments described herein were produced in an Endura®

21   Integrated Processing System, available from Applied Materials of Santa Clara,

22   California. The physical vapor deposition (sputtering in this case) process chamber is

23   capable of processing an 8 inch (200 mm) diameter silicon wafer. The substrate was a

24   silicon wafer having a silicon oxide surface coating with trenches in the surface of the

25   silicon oxide. Sputtering was carried out using a tantalum target cathode having a

1   diameter of approximately 35.3 cm (14 in.), and DC power was applied to this cathode

2   over a range from about 1 kW to about 18 kW. The substrate was placed at a distance of

3   about 25 cm (9.8 in.) from the tantalum target cathode in the case of gamma sputtering,

4   and at a distance of about 14 cm (5.5 in.)from the cathode in the case of IMP sputtering.

5   During IMP sputtering, an AC bias power ranging from about 0 W to about 400 W was

6   applied to the substrate to produce a substrate offset bias ranging from about 0 V to

7   about -100 V. The substrate offset bias attracts ions from the plasma to the substrate.

8   EXAMPLE ONE:

9   When gamma-sputtered tantalum film was produced, the film was sputtered using

10   conventional (traditional) magnetron sputtering, with rotating magnet-defined erosion

11   paths (for better uniformity and cathode utilization). Two hundred (200) mm sample

12   surfaces were sputter-deposited at a sample surface temperature of about 25°C, in argon,

13   at pressures of about 1.5 mT or less. The cathode-to-sample or "throw" distance was

14   typically about 25 cm. The DC power to the tantalum target was approximately 4 kW.

15   No substrate offset bias was used. Under these conditions, the residual film stress of the

16   tantalum film was about -1.5 x 10^{+10} dynes/cm^2.

17   EXAMPLE TWO:

18   When IMP-sputtered tantalum film was produced, a high density, inductively

19   coupled RF plasma was generated in the region between the target cathode and the

20   substrate by applying RF power to a coil (having from 1 to 3 turns) over a range from

21   about 400 kHz to about 13.56 MHZ (preferably about 2 MHZ). Two hundred (200) mm

22   sample surfaces were IMP sputter-deposited at a sample surface temperature of about

23   25°C, in argon, at pressures ranging from about 10 mT to about 60 mT. The distance

24   from the cathode to the sample was typically about 14 cm. The DC power to the

1　　　tantalum target was adjusted over a range from about 1 kW to about 8 kW (preferably,

2　　　about 1 kW to about 3 kW). The wattage to the RF power coil was adjusted over a range

3　　　from about 1.0 kW to about 5 kW (preferably, about 1.0 kW to about 3 kW). An AC

4　　　bias power ranging from about 0 W to about 500 W was used.

5　　　　　　Figure 1 shows a graph 100 of the residual film stress 101 of the tantalum film in

6　　　dynes/cm^2, as a function of the RF power 108 to the ionization coil, as illustrated by the

7　　　curve numbered 102; the pressure 110 in the sputtering chamber, as illustrated by the

8　　　curve numbered 104; and the DC power 112 to the sputtering target (cathode), as

9　　　illustrated by the curve numbered 106.

10　　　　　As indicated in graph 100, the residual stress in the deposited Ta film can be

11　　　tuned over a wide range, for example (but not by way of limitation), from about

12　　　$1.0 \times 10^{+10}$ to about $-2 \times 10^{+10}$ dynes/cm^2, and can be set at a low stress nominal value, for

13　　　example, between about $6 \times 10^{+9}$ and about $-6 \times 10^{+9}$ dynes/cm^2, a range over which the

14　　　residual stress can approach zero. At a residual stress of about $-6 \times 10^{+9}$ dynes/cm^2, by

15　　　way of example, the IMP-sputtered film residual compressive stress is a factor of three

16　　　lower than the residual compressive stress of a typical gamma-sputtered Ta film. The

17　　　process variables which affect film residual stress can be optimized to produce the

18　　　desired residual film stress in Ta films.

19　　　　　　Figures 2A and 2B show the effect of an increase in the RF power to the IMP

20　　　ionization coil, which is directly related to the amount of ion bombardment at the

21　　　tantalum film surface. Figure 2A, graph 200, shows the Ta residual film stress

22　　　in curves 201 through 206, when the power to the ionization coil is 1 kW, as a function

23　　　of process chamber argon pressure 207 and the DC power to the tantalum target 208.

24　　　Figure 2B, graph 220, shows the Ta residual film stress interior of ellipses 221 and 222,

25　　　when the power to the ionization coil is 3 kW, as a function of process chamber argon

26　　　pressure 227 and the DC power to the tantalum target 228.

1   These curves show that, with the other process values held constant, an increase

2   in RF power to the ionization coil from 1 kW to 3 kW results in an increase in the film

3   residual compressive stress. Even so, under all of the process conditions shown, the

4   residual film stress for the IMP-sputtered tantalum is less than that of a gamma-sputtered

5   tantalum film. We have concluded, then, that there is an optimum amount of ion

6   bombardment of a tantalum film surface to produce a Ta film having only minor residual

7   stress (whether compressive or in tension). Process pressure appears to have the greatest

8   effect of the variables tested. It is believed that an increase in the process pressure leads

9   to an increase in ionization within the process chamber, which leads to increased ion

10   bombardment of the depositing film surface.

11   <u>EXAMPLE THREE:</u>

12   The effect of the increase in ion bombardment of a depositing film surface, which

13   can be achieved by increasing the DC offset bias voltage of the substrate onto which the

14   film is deposited, is illustrated in Figure 3. Graph 300 shows the residual stress 311 in

15   dynes/cm^2 310 as a function of the AC bias power 320 in Watts. The corresponding

16   substrate DC offset bias voltage ranges from about 0 V to about -150 V.

17   <u>EXAMPLE FOUR:</u>

18   When tantalum nitride films are produced, the structure of the tantalum nitride

19   depends on the amount of nitrogen in the tantalum nitride compound (film). Figures 4

20   and 5 show the chemical composition and resistivity of tantalum nitride films produced

21   using gamma sputtering and IMP sputtering techniques, respectively. The chemical

22   composition (atomic nitrogen content) of the film is shown as a function of the nitrogen

23   gas flow rate to the process chamber in which the TaN_x film is produced.

1   Figure 4, graph 400, shows the nitrogen content 410 of the gamma-sputtered

2   tantalum nitride film in atomic % 413, as a function of the nitrogen flow rate 416 in

3   sccm to the process vessel. A two hundred (200) mm diameter sample surface was

4   gamma sputter-deposited at a sample surface temperature of about 25°C, in an

5   argon/nitrogen atmosphere, at a pressure of about 1.5 mT, where the argon gas feed was

6   about 15 sccm, and the nitrogen flow rate 416 was as shown on graph 400. The "throw"

7   distance between the tantalum target and the sample surface was approximately 250 mm.

8   The DC power to the tantalum target was about 4 kW.

9   In addition, graph 400 shows the resistivity 412 in μ Ω-cm 414 of the tantalum

10   nitride film as the nitrogen content 413 increases. The resistivity corresponds with the

11   change in the tantalum nitride structure, as indicated on graph 400, where 402 represents

12   β-Ta; 404 represents bcc - Ta(N); 406 represents amorphous TaN_x; and 408 represents

13   nanocrystalline fcc - TaN_x (x $\approx$ 1).

14   Figure 4 shows that when the atomic nitrogen content exceeds about 45% to

15   about 50%, the resistivity of the TaN_x film increases drastically (to above 1,000 μ Ω-cm).

16   Figure 6, graph 600, shows the residual film stress in dynes/cm^2 602 of a

17   gamma-sputtered TaN_x film, as a function of the nitrogen flow rate to the process

18   chamber in sccm 604, and as a function of the substrate temperature at the time of film

19   deposition, when the other process variables are held at the values described with

20   reference to Figure 4.

21   Curve 610 represents the TaN_x film gamma-sputtered at a substrate temperature

22   of about 25°C; Curve 612 represents the TaN_x film gamma-sputtered at a substrate

23   temperature of about 250°C, and Curve 614 represents the TaN_x film gamma-sputtered at

24   a substrate temperature of about 450°C.

1   Line 606 constructed at a nitrogen flow rate 604 of about 16 sccm, represents the

2   atomic nitrogen content in excess of which the resistivity of the TaN_x film increases

3   drastically (as illustrated in Figure 4 for a nitrogen flow rate of 16 sccm). Thus, the

4   gamma-sputtered TaN_x films having reduced residual compressive stress (in the direction

5   of arrow 608) occur at nitrogen contents at which the resistivity of the film is

6   unacceptably high (greater than about 1,000 $\mu \Omega$ - cm). Looking at the residual film

7   stress of TaN_x films having a resistivity lower than about 1,000 $\mu \Omega$ - cm, it is evident

8   that residual film stress can be reduced by increasing the substrate temperature at the

9   time of film deposition. This is in contrast with TaN_x films having a resistivity higher

10   than about 1,000 $\mu \Omega$ - cm, where the residual film stress increases when the substrate

11   temperature is higher during film deposition. Considering this unexpected result, for

12   gamma-sputtered films having a nitrogen content below about 45% - 50 %, it is

13   preferable to deposit the TaN_x film at a substrate temperature of at least about 250°C,

14   and more preferably at a substrate temperature of at least about 350°C.

15   <u>EXAMPLE FIVE:</u>

16   Figure 8 shows a graph 800 of the residual film stress of the tantalum film in

17   dynes/cm^2 804, as a function of the heater temperature in °C 806 of the substrate support

18   platen. The actual substrate temperature is approximately 50°C lower than the

19   temperature of the substrate support platen.

20   The other process conditions were as follows: The plasma source gas was 7 sccm

21   of argon, with a wafer backside gas flow of 15 sccm argon, and the process chamber

22   pressure was about 36 mT. The RF power to the ionization coil was 1.5 kW at a

23   frequency of 2 MHZ; the DC power to the tantalum target was 1 kW. No bias power

24   was applied to the substrate. However, recent experiments have shown that application

25   of a bias power sufficient to create a DC offset voltage of up to about -80 V has no

1   significant effect on the stress tuning of the films

2   There are two curves on which the film stress can be tuned. The first curve,

3   labeled 802, is representative of the β phase Ta which is deposited over a substrate

4   temperature range from about 25°C up to about 350°C. The second curve, labeled 808,

5   is representative of the bcc tantalum structure which is deposited over a substrate

6   temperature range from about 350°C through about 500°C or greater.

7   As can be observed from Figure 8, in the instance where a single tantalum film is

8   being deposited and it is desired to minimize the stress in the film (*i.e.*, to obtain a

9   relatively neutral film which is not in compression or tension), the substrate temperature

10   should be tuned within specific temperature ranges. The first temperature range is from

11   about 100°C (*i.e.*, a heater temperature of about 150°C) to about 220°C (*i.e.*, a heater

12   temperature of about 270°C). The second temperature range is from about 320°C (*i.e.*, a

13   heater temperature of about 370°C) to about 360°C (*i.e.*, a heater temperature of about

14   410°C). This presumes all other variables are held constant at the values indicated in

15   this example. Moderate stress films of $\pm$ 0.5 x 10^{10} dynes/cm^2 can be obtained over a

16   broader substrate temperature range from about 50°C to about 260°C, or from about

17   360°C to about 385°C.

18   If it is desired to create a particular tensile stress or compressive stress in the

19   tantalum film to balance the stress in other films in the film stack, the proper substrate

20   temperature can be selected.

21   EXAMPLE SIX:

22   Figure 5 graph 500 shows the nitrogen content 510 of the reactive IMP-sputtered

23   TaN$_x$ film in atomic % 513, as a function of the nitrogen flow rate in sccm 516 to the

24   process chamber. A two hundred (200) mm diameter sample (substrate) surface was

25   reactive IMP sputter-deposited at a sample surface temperature of about 25°C, in an

1   argon/nitrogen atmosphere, at a pressure of about 40 mT, where the argon gas feed was

2   about 95 sccm (80 sccm to the process chamber feed and 15 sccm to the heat exchange

3   surface), and the nitrogen flow rate 516 was as shown on graph 500. The DC power to

4   the tantalum target was about 2 kW. The RF power to the IMP induction coil was about

5   1.5 kW. No offset bias of the substrate was used.

6   In addition, graph 500 shows the resistivity 512 in μ Ω-cm 514 of the IMP-

7   sputtered TaN_x film as the atomic nitrogen content 513 increases. The resistivity

8   corresponds with the change in the tantalum nitride structure, as indicated on Graph 500,

9   where 502 represents β-Ta; 504 represents bcc - Ta(N); 506 represents amorphous TaN_x;

10   and 508 represents nanocrystalline fcc - TaN_x (x $\approx$1).

11   Figure 5 also shows that, when the atomic nitrogen content exceeds about 45%,

12   the resistivity of the TaN_x film increases drastically (to above 1,000 μ Ω-cm).

13   Figure 7, graph 700, shows the residual film stress in dynes/cm^2 702 of an IMP-

14   sputtered TaN_x film, as a function of the nitrogen flow rate to the process chamber in

15   sccm 704, for deposition on a substrate at a temperature of about 25°C, when the other

16   process variables are held at the values described with reference to Figure 5.

17   Line 706, constructed at a nitrogen flow rate 704 of about 14 - 16 sccm,

18   represents the atomic nitrogen content in excess of which the resistivity of the TaN_x film

19   increases drastically (as illustrated in Figure 5). We discovered that for IMP-sputtered

20   TaN_x films, in contrast with the gamma-sputtered films, it is possible to produce a film

21   having reduced residual stress at the lower nitrogen contents, where an acceptable

22   resistivity can be obtained. Further, the IMP-sputtered TaN_x film residual stress appears

23   to remain relatively unaffected by an increase in the nitrogen content over the nitrogen

24   content range represented by the nitrogen flow rates illustrated in Figure 7 (up to about

25   60 atomic % nitrogen, based on Figure 5).

1   By depositing the TaN$_x$ film using the IMP sputtering method which provides

2   increased bombardment of the depositing film surface (over that obtained by the gamma

3   sputtering method), it is possible to produce a TaN$_x$ film having both an acceptable

4   resistivity and reduced residual film stress. This is because the IMP-sputtered TaN$_x$ film

5   stress remains relatively unchanged with increasing nitrogen content (in comparison with

6   gamma-sputtered TaN$_x$ film stress, which is strongly dependent on the nitrogen content

7   of the film in the region where the film resistivity is acceptable).

8   V.   THE METHOD OF IMPROVING BARRIER PERFORMANCE OF Ta AND
9   TaN$_x$ FILMS

10   We have also discovered that Ta and TaN$_x$ films deposited using physical vapor

11   deposition techniques at a temperature which falls within a range of about 350°C to

12   about 600°C have improved diffusion barrier properties over those deposited at room

13   temperature, as discussed in Examples Seven and Eight, which follow.

14   EXAMPLE SEVEN:

15   Ta films were produced using IMP sputter deposition and gamma sputter

16   deposition. Process conditions for the IMP sputter deposition were as follows: The

17   plasma source gas was 7 sccm argon, with a wafer backside heat transfer gas flow rate of

18   15 sccm argon, and the process chamber pressure was 35 mT. The RF power to the

19   ionization coil was 1.5 kW at a frequency of 2 MHZ; the DC power to the tantalum

20   target was 1 kW. No bias power was applied to the substrate.

21   Process conditions for the gamma sputter deposition were as follows: The

22   plasma source gas was 7 sccm of argon, with a wafer backside gas flow of 15 sccm

23   argon, and the process chamber pressure was about 1 - 2 mT. The DC power to the

24   tantalum target was 4 kW.

1   Ta barrier films were deposited using each deposition method at room

2   temperature (25°C) and using substrate heater temperatures of 350°C and 500°C, which

3   result in substrate temperatures of about 300°C and 450°C, respectively.

4   The diffusion barrier performance of the Ta films was characterized using

5   electrical testing. MOS capacitors having silicon/silicon dioxide/tantalum/copper

6   structures (wherein the tantalum was deposited using the present method for improving

7   barrier performance) were subjected to Bias-Temperature Stress (BTS) testing at 275°C

8   and 2 MV/cm. Current through the silicon dioxide was measured during BTS. Diffusion

9   of copper through the barrier material into the silicon dioxide leads to catastrophic oxide

10   failure. The median time-to-failure (MTTF) was used to compare different barrier

11   material performance, as shown in Table One, below.

12   Table One. Barrier Performance of Ta Films

Film Thickness	Substrate Temperature During Deposition	MTTF (n = 20)
100 Å	25°C	2 hours
100 Å	450°C	10 hours
200 Å	25°C	4 hours
200 Å	300°C	6 hours

18   n = the number of samples tested to provide an average MTTF.

19   As the data in Table One indicate, increases in deposition temperature led to

20   significant improvement in barrier performance.

21   Deposition at elevated temperatures results in an increase in the surface

22   diffusivity of adatoms, as well as enhanced grain boundary coalescence. As a result, the

23   film density increases, which in turn improves the barrier performance against copper

24   diffusion. Our results showed that Ta deposited at 500°C was a bcc phase, and Ta

1   deposited at room temperature is a β phase. The bcc phase has a higher density than the

2   β phase, and therefore provides a better barrier layer.

3   EXAMPLE EIGHT:

4   TaN$_x$ films were produced using IMP sputter deposition and gamma sputter

5   deposition. Process conditions for the IMP sputter deposition were as follows: The

6   plasma source gas was 7 sccm argon in combination with 16 sccm nitrogen, with a wafer

7   backside gas flow of 15 sccm argon, and the process chamber pressure was 39 mT. The

8   RF power to the ionization coil was 1.5 kW at a frequency of 2 MHZ; the DC power to

9   the tantalum target was 1 kW. Samples were produced without bias power applied to the

10   substrate and with about 275 W applied to the substrate support platen, to produce a DC

11   offset voltage of about -70 V to about -80 V. The same results were obtained with and

12   without applied bias.

13   Process conditions for the gamma sputter deposition were as follows: The

14   plasma source gas was 7 sccm of argon in combination with 14 sccm of nitrogen, with a

15   wafer backside gas flow of 15 sccm argon, and the process chamber pressure was about

16   1 - 2 mT. The DC power to the tantalum target was 4 kW.

17   TaN$_x$ barrier films having a thickness of 100 Å were deposited on silicon

18   substrates using each deposition method at room temperature and using a substrate heater

19   temperature of 450°C, which resulted in a substrate temperature of about 400°C. A

20   copper layer having a thickness of about 1500 Å was then deposited over the TaN$_x$

21   barrier layer. The film stacks were then annealed under vacuum at a heater temperature

22   of about 600°C for 30 minutes.

23   Sheet resistivity of the film stacks was measured before and after a vacuum

24   annealing in an argon ambient. Film stacks having barrier layers which failed during

25   annealing exhibited etch pits where the copper had diffused through the TaN$_x$ layer and

1   reacted with the underlying silicon. The etch pits were observed visually.

2   The barrier performance for the TaN$_x$ films is shown in Table Two, below.

3   Table Two. Barrier Performance of TaN$_x$ Films

Deposition Method	Substrate Temperature During Deposition	Barrier Performance (Etch Pit Test)
Gamma	25°C	Fail
Gamma	400°C	Pass
IMP	25°C	Fail
IMP	400°C	Pass

9   As shown in Table Two, the devices incorporating the TaN$_x$ barrier layers

10   deposited at a substrate temperature of 400°C passed, whereas those deposited at 25°C

11   failed, regardless of the deposition method. A 30-minute annealing at a heater

12   temperature of about 600°C failed to improve the barrier performance of the TaN$_x$ films

13   deposited at 25°C. As with the Ta films described in Example Five, an increase in

14   deposition temperature again led to significant improvement in barrier performance for

15   the tantalum nitride films.

16   The above described preferred embodiments are not intended to limit the scope of

17   the present invention, as one skilled in the art can, in view of the present disclosure,

18   expand such embodiments to correspond with the subject matter of the invention claimed

19   below.

CLAIMS

We claim:

1   1.   A Ta film tuned to have a residual film stress ranging between about $1.0 \times 10^{+10}$
2   dynes/cm² and about $-2 \times 10^{+10}$ dynes/cm².

1   2.   The Ta film of Claim 1, wherein said Ta film was sputter-deposited.

1   3.   The Ta film of Claim 2, wherein said Ta film was IMP sputter-deposited.

1   4.   The Ta film of Claim 1, wherein said film residual stress ranges between about
2   $6 \times 10^{+9}$ dynes/cm² and about $-6 \times 10^{+9}$ dynes/cm².

1   5.   The Ta film of Claim 1, wherein a crystalline structure of said tantalum film is
2   bcc Ta.

1   6.   The Ta film of Claim 1, wherein a crystalline structure of said tantalum film is β
2   Ta.

1   7.   A TaN$_x$ film, where $0 < x \leq 1.5$, tuned to have a residual film stress ranging
2   between about $1.0 \times 10^{+10}$ dynes/cm² and about $-2 \times 10^{+10}$ dynes/cm².

1   8.   The TaN$_x$ film of Claim 7, wherein said TaN$_x$ film was sputter-deposited.

1   9.   The TaN$_x$ film of Claim 8, wherein said TaN$_x$ film was reactive IMP sputter-
2   deposited.

1   10.   The TaN$_x$ film of Claim 7, wherein said film residual stress ranges between about

2   6 x 10^{+9} dynes/cm^2 and about -6 x 10^{+9} dynes/cm^2.

1   11.   The TaN$_x$ film of Claim 7, wherein said film resistivity is less than about 1,000

2   μ Ω-cm and said film stress ranges between about 6 x 10^{+9} dynes/cm^2 and about -6 x 10^{+9}

3   dynes/cm^2.

1   12.   The TaN$_x$ film of Claim 11, wherein said film comprises more than about 30

2   atomic % nitrogen.

1   13.   The TaN$_x$ film of Claim 12, wherein said film comprises and less than about 60%

2   nitrogen.

1   14.   A method of tuning the residual film stress of a Ta film, wherein said residual

2   stress is tuned by controlling the amount of ion bombardment of the depositing film

3   surface.

1   15.   The method of Claim 14, wherein said Ta film is deposited using a sputtering

2   technique.

1   16.   The method of Claim 15, wherein said Ta film is deposited using IMP sputtering.

1   17.   The method of Claim 15, wherein said tantalum film comprises bcc Ta.

1   18. The method of Claim 15, wherein a crystalline structure of said tantalum film

2   is β Ta.

1   19. A method of tuning the residual film stress of a Ta film by adjustment of a film

2   deposition process variable selected from the group consisting of process chamber

3   pressure, substrate DC offset bias voltage, power to a sputtering target, power to an

4   ionization coil, substrate temperature, or a combination thereof.

1   20. The method of Claim 19, wherein said residual film stress is tuned to range

2   between about $1 \times 10^{+10}$ dynes/cm^2 and about $-2 \times 10^{+10}$ dynes/cm^2.

1   21. A method of tuning the residual film stress of a Ta film subsequent to deposition,

2   wherein said treatment is selected from the group consisting of ion bombardment,

3   annealing, and combinations thereof.

1   22. The method of Claim 21, wherein said residual film stress is tuned to range

2   between about $1 \times 10^{+10}$ dynes/cm^2 and about $-2 \times 10^{+10}$ dynes/cm$_2$.

1   23. The method of Claim 21, wherein said method of tuning is ion bombardment.

1   24. The method of Claim 21, wherein said method of tuning is annealing, and

2   wherein said annealing is carried out at a temperature of at least about 25°C.

1   25. The method of Claim 24, wherein said temperature is at least about 250°C.

1   26. The method of Claim 25, wherein said temperature is at least about 350°C.

1   27.   A method of tuning the residual film stress of a TaN_x film, wherein said residual

2   stress is tuned by controlling the amount of ion bombardment of the depositing film

3   surface and where $0 < x \leq 1.5$.

1   28.   The method of Claim 27, wherein said TaN_x film is deposited using a sputtering

2   technique.

1   29.   The method of Claim 28, wherein said TaN_x film is deposited using reactive IMP

2   sputtering.

1   30.   The method of Claim 29, wherein said TaN_x film comprises at least about 30

2   atomic % nitrogen.

1   31.   The method of Claim 30, wherein said nitrogen content is less than about 60

2   atomic % nitrogen.

1   32.   A method of tuning the residual film stress of a TaN_x film by adjustment of a film

2   deposition process variable selected from the group consisting of process chamber

3   pressure, substrate DC offset bias voltage, power to a sputtering target, power to an

4   ionization coil, substrate temperature, or a combination thereof.

1   33.   The method of Claim 32, wherein said residual film stress is tuned to range

2   between about $1 \times 10^{+10}$ dynes/cm^2 and about $-2 \times 10^{+10}$ dynes/cm^2.

1   34.   A method of tuning the residual film stress of a TaN_x film subsequent to

2   deposition, wherein said treatment is selected from the group consisting of ion

3   bombardment, annealing, and combinations thereof.

1   35.   The method of Claim 34, wherein said residual film stress is tuned to range

2   between about $1 \times 10^{+10}$ dynes/cm^2 and about $-2 \times 10^{+10}$ dynes/cm$_2$.

1   36.   The method of Claim 34, wherein said method of tuning is ion bombardment.

1   37.   The method of Claim 34, wherein said method of tuning is annealing, and

2   wherein said annealing is carried out at a temperature of at least about 25°C.

1   38.   The method of Claim 37, wherein said temperature is at least about 250°C.

1   39.   The method of Claim 38, wherein said temperature is at least about 350°C.

1   40.   In a physical vapor deposition method of depositing an improved Ta or TaN_x

2   barrier layer in a semiconductor interconnect structure, a method of improving the barrier

3   performance of Ta or TaN_x, wherein the improvement comprises depositing said Ta or

4   TaN_x barrier layer at a substrate temperature of at least 300°C, wherein $0 < x \le 1.5$.

1   41.   The method of Claim 40, wherein said barrier layer is deposited at a substrate

2   temperature within the range of about 300°C to about 600°C.

1   42.   The method of Claim 41, wherein said barrier layer is deposited at a substrate

2   temperature within the range of about 300°C to about 500°C.

1   43.   The method of Claim 40, wherein said barrier layer is deposited using gamma

2   sputter deposition.

1   44.   The method of Claim 40, wherein said barrier layer is deposited using IMP

2   sputter deposition.

1   45.   The method of Claim 40, wherein subsequent to the deposition of said barrier

2   layer, a layer of copper is deposited thereover.

1   46.   The Ta film of Claim 1, wherein the substrate temperature during deposition of

2   said Ta film is at least 300°C.

1   47.   The Ta film of Claim 2, wherein the substrate temperature during deposition of

2   said Ta film is at least 300°C.

1   48.   The Ta film of Claim 3, wherein the substrate temperature during deposition of

2   said Ta film is at least 300°C.

1   49.   The TaN_x film of Claim 7, wherein the substrate temperature during deposition of

2   said TaN_x film is at least 300°C.

1   50.   The TaN_x film of Claim 8, wherein the substrate temperature during deposition of

2   said TaN_x film is at least 300°C.

1   51.   The TaN$_x$ film of Claim 9, wherein the substrate temperature during deposition of

2   said TaN$_x$ film is at least 300°C.

1   52.   The method of Claim 3, wherein the substrate temperature during said Ta film

2   deposition is selected to be within a range of about 100°C to about 220°C or within a
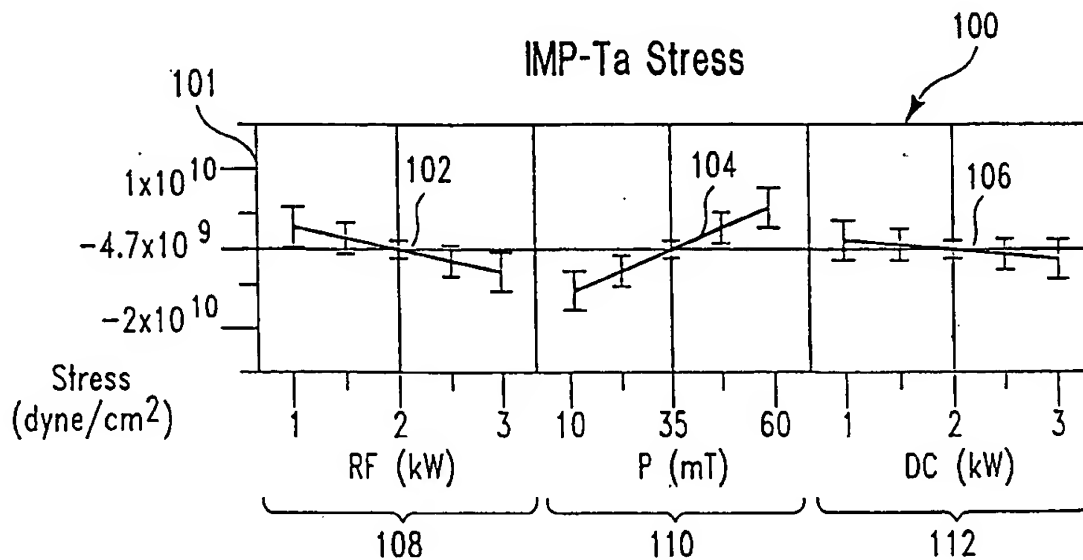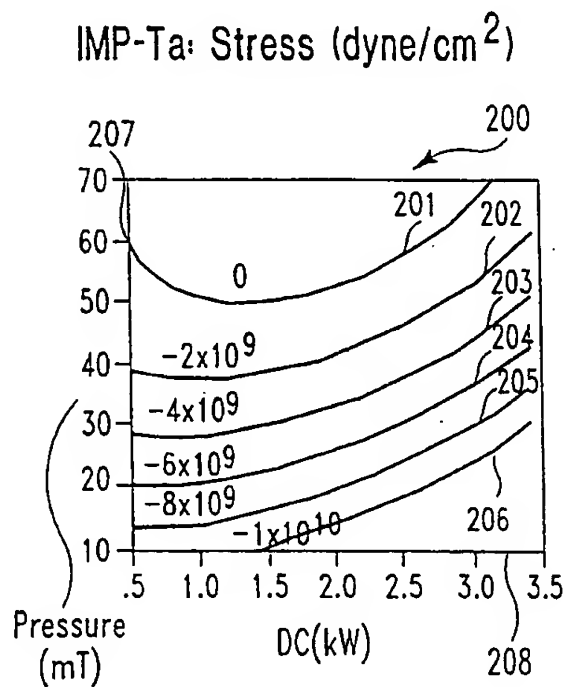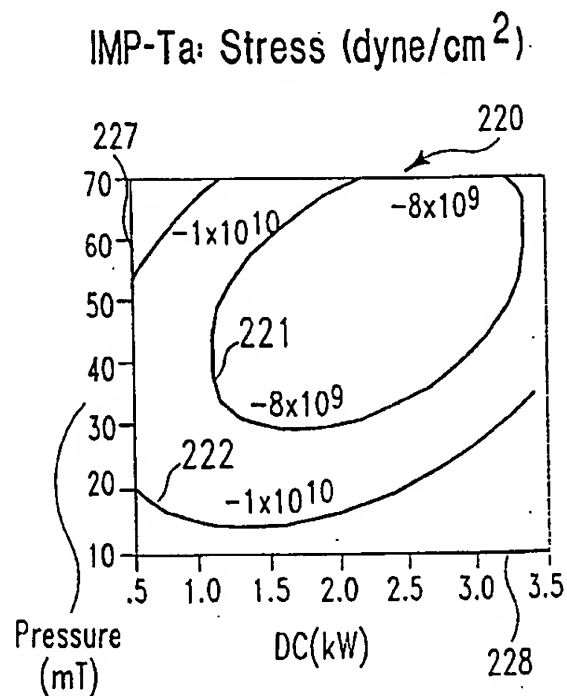
3   range of about 360°C to about 410°C.

1   53.   The method of Claim 9, wherein the substrate temperature during said TaN$_x$ film

2   deposition is selected to be within a range of about 100°C to about 220°C or within a

3   range of about 360°C to about 410°C.

IMP-Ta Stress

FIG. 1

IMP-Ta: Stress (dyne/cm^2)

IMP-Ta: Stress (dyne/cm^2)

FIG. 2A

FIG. 2B

2 / 5

IMP-Ta Stress (dyne/cm^2)   300

Bias (W)

FIG. 3

3 / 5

Gamma TaN_x Composition

FIG. 4

IMP-TaN_x Composition

FIG. 5

Gamma TaN$_x$ Film Stress

FIG. 6

IMP-TaN$_x$ Film Stress

FIG. 7
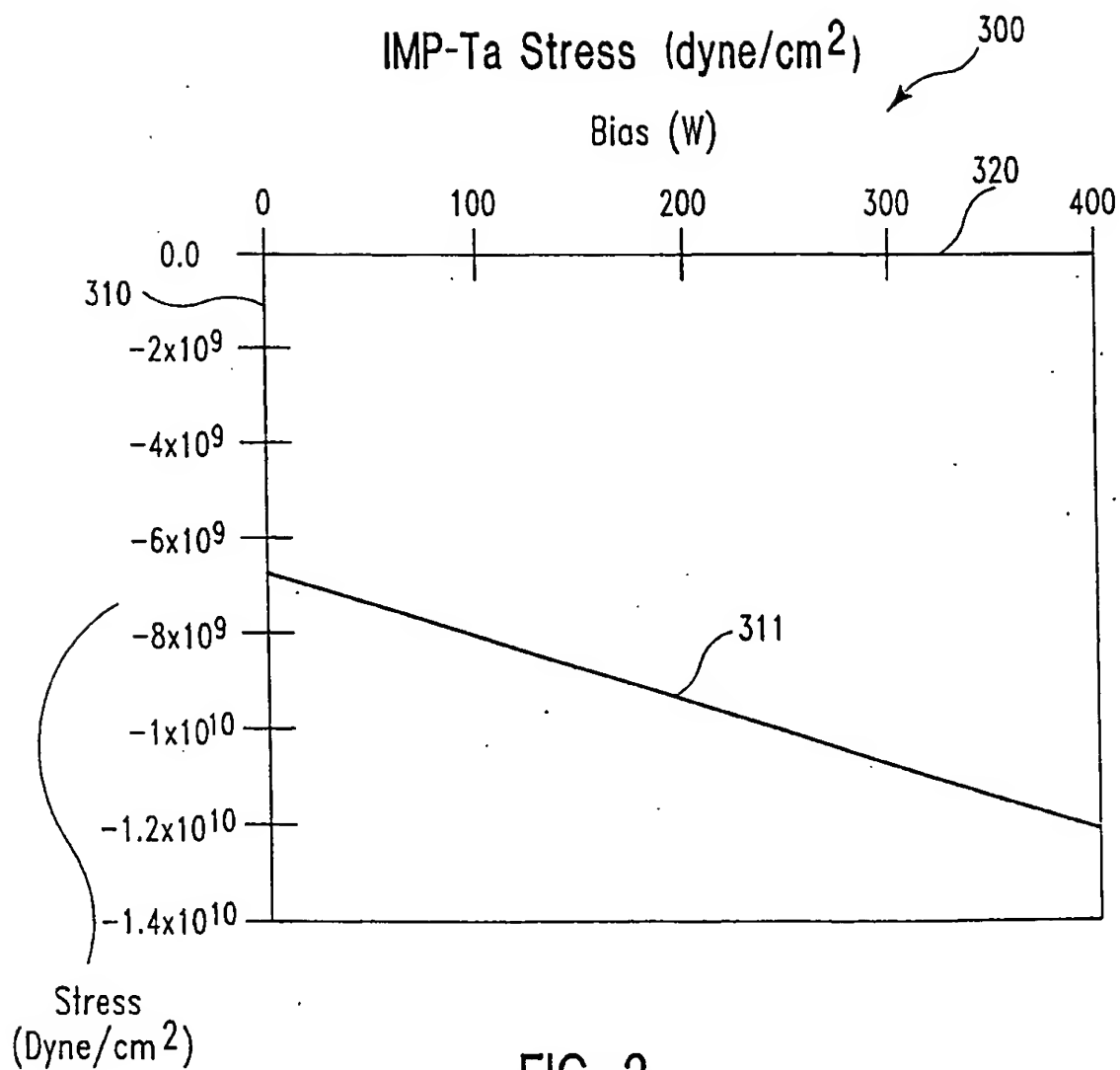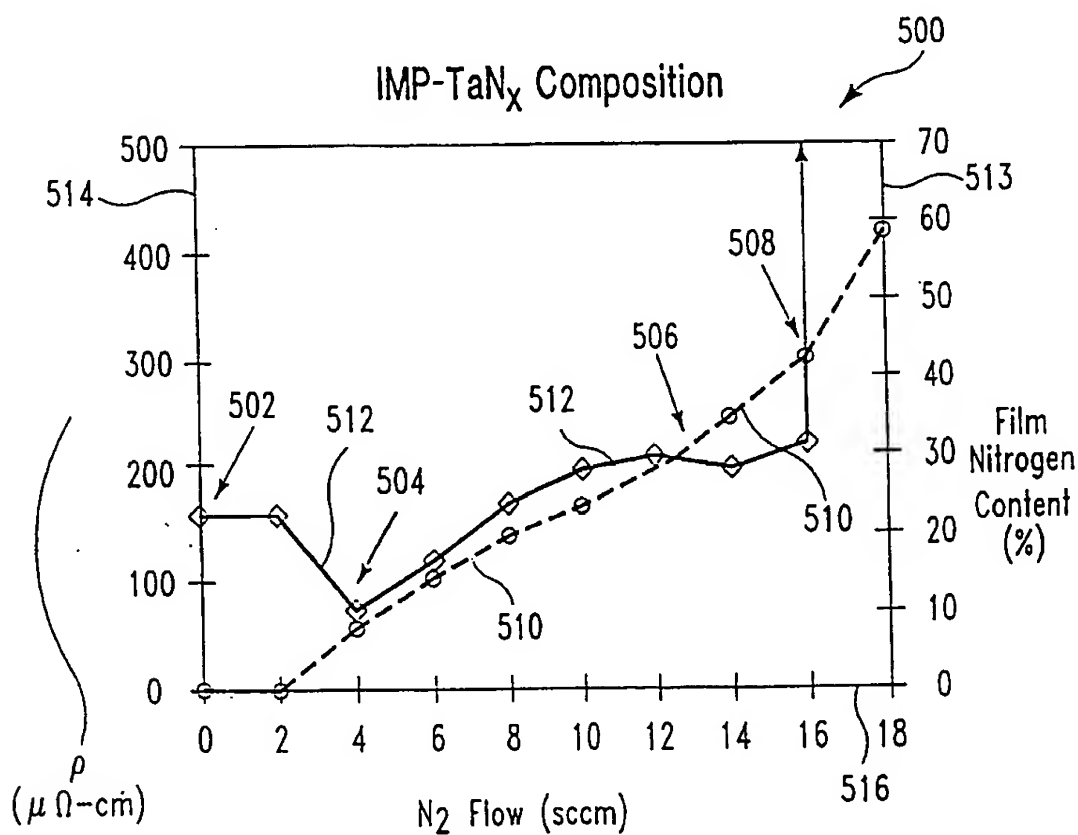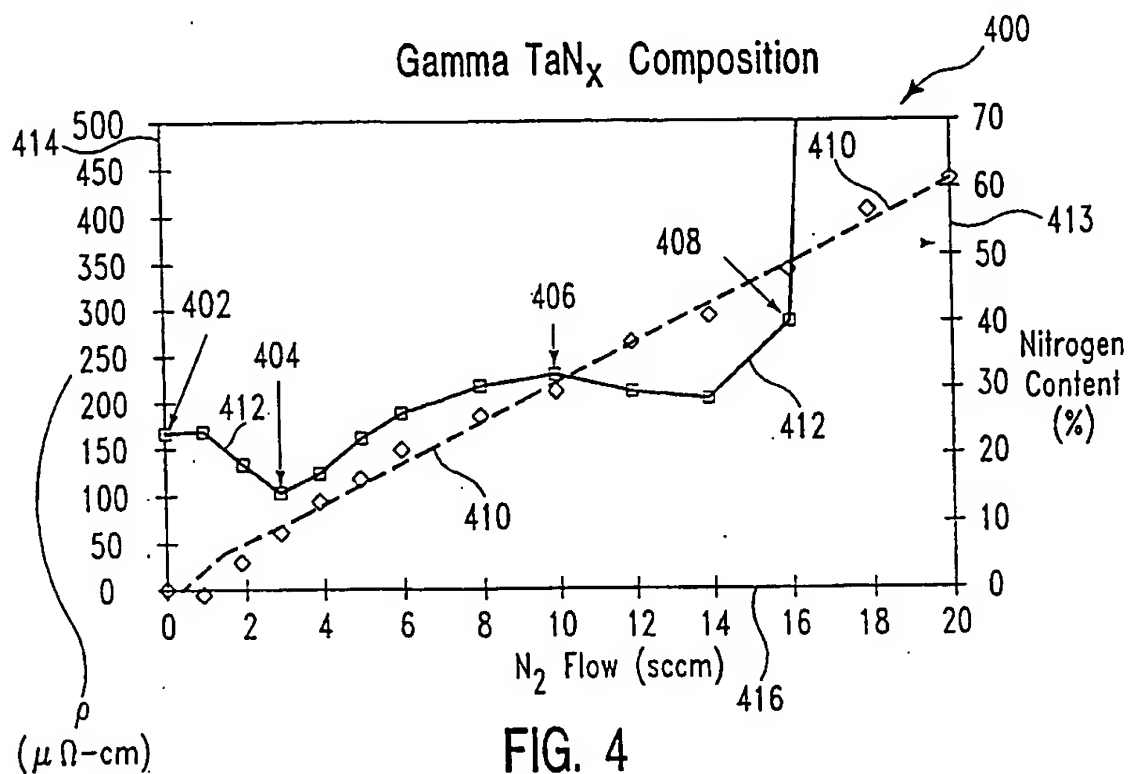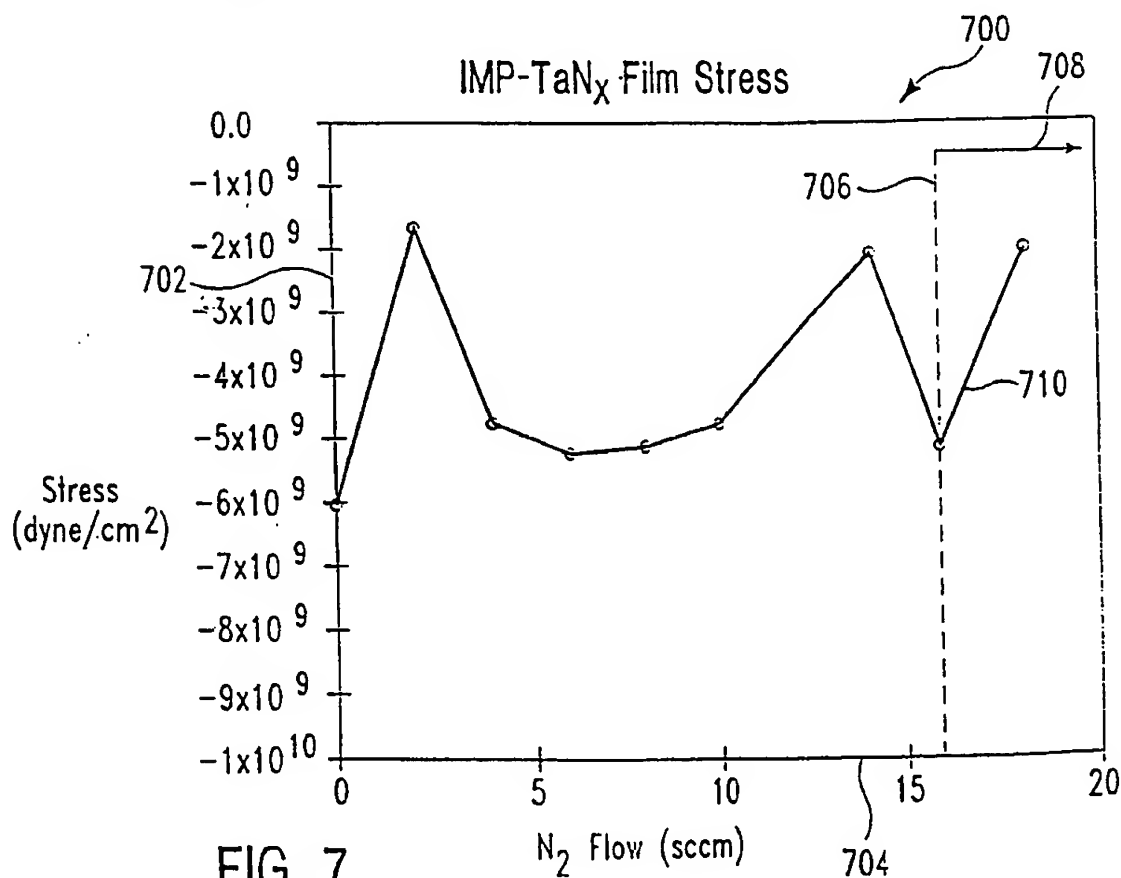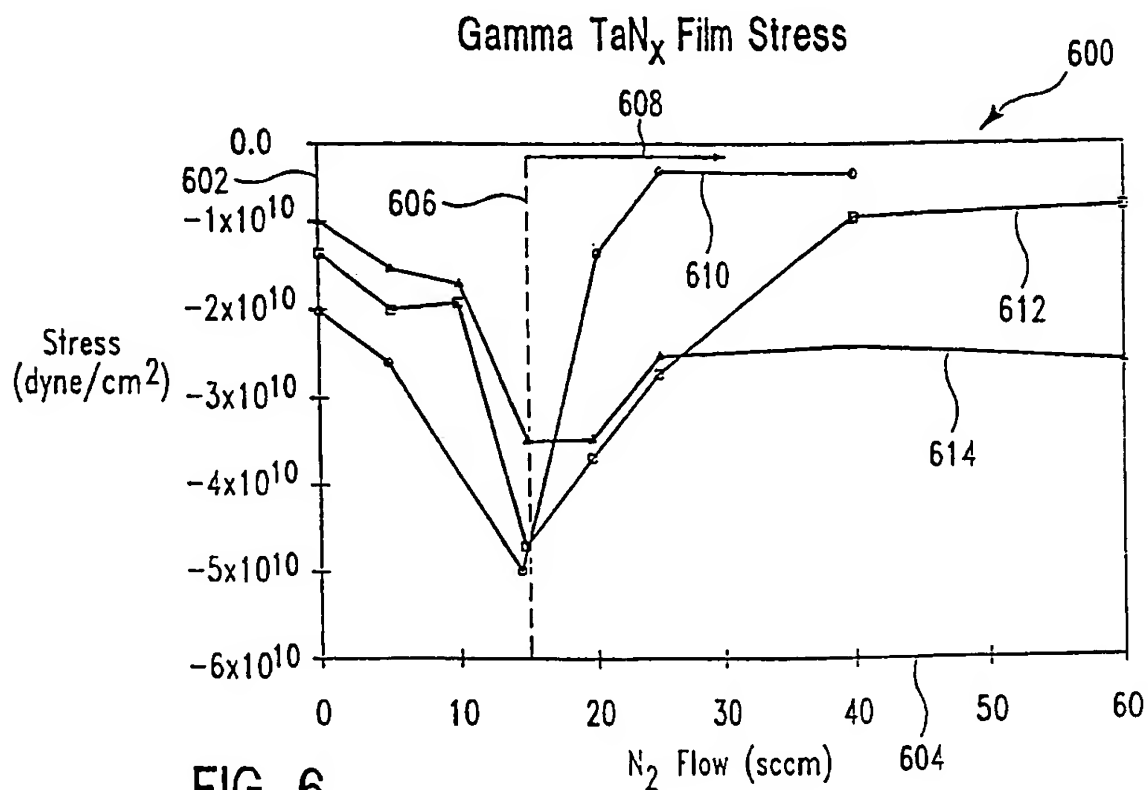
IMP-Ta Stress v. Temperature

FIG. 8